IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of : **Confirmation No. 6167**

Takatsugu TAKAMURA et al. : Attorney Docket No. 2003_1757A

Serial No. 10/726,652 : Group Art Unit 1711

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PRODUCTION METHOD OF BIODEGRADABLE PLASTIC AND APPARATUS FOR USE IN PRODUCTION THEREOF

RULE 132 DECLARATION

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Toshinobu Yoshihara , the undersigned, a citizen of Japan, residing at 6-1, Todaimon 1-chome, Midori-ku, Saitama-shi, Saitama , Japan, do hereby declare:

1. That I am a development collaborator of the above-identified application.

2. That I graduated from Nikko High School on March , 1978 .

3. 1996 to present

NBC CO., LTD.

President of NBC.CO.,LTD and developing of Biodegradable plastic and apparatus, and au pair cornsheet.

1979 to 1995

NIPPON TRAVEL AGENCY CO.,LTD.

4. There are no publications, awards, or other distinguishing professional recognizations.

5. That in order to show the unexpected properties of the combined catalyst system of zinc chloride and stannous chloride of this invention over the individual use of such catalysts,

I have under my control and direction conducted the following experiments. The experiments compare the manufacture of polylactic acid from lactic acid monomers by the direct condensation method using the combined catalyst system of this invention with the same catalysts used alone. The particulars and results of the experiments are set forth hereinbelow.

Example 1

The catalyst used in this Example was 0.2% of the zinc chloride and 0.5% of the stannous chloride according to the invention.

90% L-lactic-acid monomer (7.5 kg) was supplied to the reaction apparatus shown in Fig. 3 of this application through the inlet 5 of the main body 1a. The monomers were mixed slowly with mixing device 4, while heating at 100 to 150°C by the heating device 3, and dehydrated over about 5 hours under a vacuum using a decompression device 2 which was applied at -0.20 to -0.05 MPa (20 to 5 mmHg). The degree of vacuum became high in the early stage of dehydration, and became low in the second half.

Then, after returning to normal pressure, the catalyst (0.2% of the zinc chloride and 0.5% of the stannous chloride) was added through inlet 5. The direct polycondensation reaction was carried out for 10 hours at a churning speed of 100 rpm, a temperature of 180°C, and a degree of vacuum -0.08 to -0.05 MPa (8 to 1 mm Hg). The degree of vacuum became high in the early stage of the reaction, and became low in the second half. The terminal point of the reaction was determined by the amount of emergence of steam and speed change which were discharged from vent 6, as detected by a sensor for water vapor 13, and caught by a measuring device for water vapor 14, and by pressure reducing unit 2. The amount of emergence of steam and speed change became high in the early stage of the reaction, and became low in the second half.

Next, the screw shaft 9 was pulled up within discharge cylinder 7 to open the product outlet 8 and polylactic acid was taken out from there. The weight average molecular weight of the obtained polylactic acid was 120,000.

Example 2

The catalyst used in this Example was 0.1% of the zinc chloride and 0.4% of the stannous chloride according to the invention.

The same reaction was carried out according to Example 1, except that the catalyst used was 0.1% of the zinc chloride and 0.4% of the stannous chloride. The same terminal point of the reaction was achieved after the direct polycondensation reaction was carried out for 12 hours. The amount of average molecular weight of the obtained polylactic acid was 110,000.

Example 3

The catalyst used in this Example was 0.5% of the stannous chloride alone.

The same reaction was carried out according to Example 1, except that the catalyst used was 0.5% of stannous chloride alone. The same terminal point of the reaction was achieved after the direct polycondensation reaction was carried out for 18 hours. The amount of average molecular weight of the obtained polylactic acid was 100,000.

Example 4

The catalyst used in this Example was 0.5% of the zinc chloride alone.

The same reaction was carried out according to Example 1, except that the catalyst used was 0.5% of the zinc chloride was used. The same terminal point of the reaction was achieved after the direct polycondensation reaction was carried out for 20 hours. The amount of average molecular weight of the obtained polylactic acid was 80,000.

The following Table summarizes the results of the experiments of Examples 1-4.

Example	Catalyst (weight %)	Weight average molecular weight	Reaction time(h)	Polycondensation temperature(°C)	Degree of vacuum(MPa)
Example 1	Zinc chloride 0.2 Stannous chloride 0.5	120,000	10	180	-0.05 ~-0.08
Example 2	Zinc chloride 0.1 Stannous chloride 0.4	110,000	12	180	-0.05 ~-0.08
Example 3	Stannous chloride 0.5	100,000	18	180	-0.05 ~-0.08
Example 4	Zinc Chloride 0.5	80,000	20	180	-0.05 ~-0.08

In summary, it is my opinion that the foregoing experiments demonstrate that the unexpectedly superior results of the combined catalyst system of zinc chloride and stannous chloride of this invention. The combined catalyst system of this invention enables the manufacture of polylactic acid having a higher weight average molecular weight in a reduced reaction time in comparison to using the same individual catalysts. Such unexpected results were not obvious from the prior art at the time of this invention.

I further declare that all statements made herein of my own knowledge are true and all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

Date: July 7, 2006

吉原利宣

(Signature of Declarant)